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NEWS 4 DEC 18 CA/CAPplus patent kind codes updated
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NEWS 6 DEC 18 MEDLINE updated in preparation for 2007 reload
NEWS 7 DEC 27 CA/CAPplus enhanced with more pre-1907 records
NEWS 8 JAN 08 CHEMLIST enhanced with New Zealand Inventory of Chemicals
NEWS 9 JAN 16 CA/CAPplus Company Name Thesaurus enhanced and reloaded
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NEWS 11 JAN 16 WPIDS/WPINDEX/WPIX enhanced with IPC 8 reclassification data
NEWS 12 JAN 22 CA/CAPplus updated with revised CAS roles
NEWS 13 JAN 22 CA/CAPplus enhanced with patent applications from India
NEWS 14 JAN 29 PHAR reloaded with new search and display fields
NEWS 15 JAN 29 CAS Registry Number crossover limit, increased to 300,000 in
multiple databases
NEWS 16 FEB 15 PATDPASPC enhanced with Drug Approval numbers
NEWS 17 FEB 15 RUSSIAPAT enhanced with pre-1994 records
NEWS 18 FEB 23 KOREAPAT enhanced with IPC 8 features and functionality
NEWS 19 FEB 26 MEDLINE reloaded with enhancements
NEWS 20 FEB 26 EMBASE enhanced with Clinical Trial Number field
NEWS 21 FEB 26 TOXCENTER enhanced with reloaded MEDLINE
NEWS 22 FEB 26 IFICDB/IFIPAT/IFIUDB reloaded with enhancements
NEWS 23 FEB 26 CAS Registry Number crossover limit increased from 10,000
to 300,000 in multiple databases
NEWS 24 MAR 15 WPIDS/WPIX enhanced with new FRAGHITSTR display format
NEWS 25 MAR 16 CASREACT coverage extended

NEWS EXPRESS NOVEMBER 10 CURRENT WINDOWS VERSION IS V8.01c, CURRENT
MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
AND CURRENT DISCOVER FILE IS DATED 25 SEPTEMBER 2006.

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FILE 'BIOSIS' ENTERED AT 12:24:26 ON 17 MAR 2007

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=> s hops

L1 6517 HOPS

=> s hopd extract

L2 0 HOPD EXTRACT

=> s hops extract

L3 248 HOPS EXTRACT

=> s humulone or humulon

L4 935 HUMULONE OR HUMULON

=> s dihydrohumulone or dihydrohumulon

L5 6 DIHYDROHUMULONE OR DIHYDROHUMULON

=> s isoalpha acid

L6 27 ISOALPHA ACID

=> s isohumulone or isocohumulone or isoadhumulone

L7 689 ISOHUMULONE OR ISOCOHUMULONE OR ISOADHUMULONE

=> s dihydroisohumulone or dihydroisocohumulone or dihydroisoadhumulone

L8 31 DIHYDROISOHUMULONE OR DIHYDROISOCOHUMULONE OR DIHYDROISOADHUMULONE

=> s L3 and L7

L9 28 L3 AND L7

=> dup rem L9

PROCESSING COMPLETED FOR L9

L10 28 DUP REM L9 (0 DUPLICATES REMOVED)

=> s L10 and L8

L11 2 L10 AND L8

=> s L7 and L8
L12 29 L7 AND L8

=> dup rem L12
PROCESSING COMPLETED FOR L12
L13 18 DUP REM L12 (11 DUPLICATES REMOVED)

=> s L10 and (AY<2004 or PY<2004 or PRY<2004)
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
2 FILES SEARCHED...
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
L14 27 L10 AND (AY<2004 OR PY<2004 OR PRY<2004)

=> s L13 and (AY<2004 or PY<2004 or PRY<2004)
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
2 FILES SEARCHED...
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
'2004' NOT A VALID FIELD CODE
L15 10 L13 AND (AY<2004 OR PY<2004 OR PRY<2004)

=> d L13 1-18 ibib abs

L13 ANSWER 1 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2007:197601 CAPLUS
TITLE: Protein kinase modulation by hops and Acacia products
INVENTOR(S): Tripp, Matthew L.; Babish, John G.; Bland, Jeff; Hall,
Amy Jennae; Konda, Veera; Desai, Anu
PATENT ASSIGNEE(S): Metaproteomics, LLC, USA
SOURCE: PCT Int. Appl., 161pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2007021694	A2	20070222	WO 2006-US30920	20060809
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW			
RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
US 2007042063	A1	20070222	US 2006-501393	20060809
PRIORITY APPLN. INFO.:			US 2005-706984P	P 20050809
			US 2005-748931P	P 20051209
AB	Botanical compds. to modulate protein kinase activity are disclosed. The compds. and methods disclosed also inhibit expression of COX-2, inhibit synthesis of prostaglandins selectively in target cells, and inhibit inflammatory response selectively. The compns. contain at least one			

fraction isolated or derived from hops or Acacia.

L13 ANSWER 2 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2006:579641 CAPLUS

DOCUMENT NUMBER: 145:51071

TITLE: Curcuminoid compositions exhibiting synergistic inhibition of the expression and/or activity of cyclooxygenase-2

INVENTOR(S): Babish, John G.; Howell, Terrance M.; Parcioretty, Linda M.

PATENT ASSIGNEE(S): Metaproteomics, LLC, USA

SOURCE: PCT Int. Appl., 36 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2006062681	A1	20060615	WO 2005-US41020	20051114
WO 2006062681	A9	20060803		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM			
US 2005129791	A1	20050616	US 2004-988393	20041113
PRIORITY APPLN. INFO.:			US 2004-988393	A 20041113
			US 2001-335062P	P 20011026
			US 2002-282236	A1 20021025

AB A novel formulation is provided that serves to inhibit the inflammatory response in animals. The formulation comprises, as a first component an effective amount of a curcuminoid species and an effective amount of a second component selected from the group consisting of an alpha-acid species, e.g., humulone, cohumulone, isohumulone, hulupone, etc., or a beta-acid species, such as lupulone, colupulone, adlupulone, etc., or derivs. thereof. The composition provides synergistic anti-inflammatory effects in response to phys. or chemical injury or abnormal immune stimulation due to a biol. agent or unknown etiol. Thus, a lotion containing 0.1% curcuminoids and 0.5% humulone or lupulone was prepared and applied to affected areas of patients who have exhibited acne rosacea or psoriasis.

REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 3 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:963807 CAPLUS

DOCUMENT NUMBER: 143:253900

TITLE: Synergistic anti-inflammatory compositions comprising an isoalpha acid and a reduced isoalpha acid from hops

INVENTOR(S): Babish, John G.; Tripp, Matthew L.; Bland, Jeffrey S.

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 21 pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005192356	A1	20050901	US 2004-789814	20040227
AU 2005219387	A1	20050915	AU 2005-219387	20050226
CA 2557676	A1	20050915	CA 2005-2557676	20050226
WO 2005084680	A1	20050915	WO 2005-US6216	20050226
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
EP 1718313	A1	20061108	EP 2005-723895	20050226
R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, FI, RO, CY, TR, BG, CZ, EE, HU, PL, SK, IS			
PRIORITY APPLN. INFO.:			US 2004-789814	A 20040227
			WO 2005-US6216	W 20050226

OTHER SOURCE(S): MARPAT 143:253900

AB The invention provides a composition comprising a reduced isoalpha acid (RIAA), selected from dihydroisohumulone, dihydroisocohumulone and dihydroadhumulone, and isoalpha acid (IAA), selected from isohumulone, isocohumulone, and isoadhumulone, isolated from hops, wherein the RIAA and IAA are in a ratio of about 3:1 to about 1:10. The invention also provides a method of reducing inflammation by administering a composition comprising a reduced isoalpha acid (RIAA) and isoalpha acid (IAA) isolated from hops, wherein the RIAA and IAA are in a ratio of about 3:1 to about 1:10. For example, synergy of PGE2 inhibition produced by four combinations of RIAA and IAA (3:1, 3:2, 1:1 and 1:10, resp.) was demonstrated in Raw 264.7 cells. Particularly relevant synergy occurred at the 1:1 and 1:10 RIAA/IAA ratios, at RIAA concns. <0.58 µg/mL and RIAA concns. >0.31 µg/mL.

L13 ANSWER 4 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN DUPLICATE 1

ACCESSION NUMBER: 2005:78545 CAPLUS

DOCUMENT NUMBER: 142:315625

TITLE: Photooxidative degradation of beer bittering principles: A key step on the route to lightstruck flavor formation in beer

AUTHOR(S): Huvaere, Kevin; Andersen, Mogens L.; Skibsted, Leif H.; Heyerick, Arne; De Keukeleire, Denis

CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, Laboratory of Pharmacognosy and Phytochemistry, Ghent University, Ghent, B-9000, Belg.

SOURCE: Journal of Agricultural and Food Chemistry (2005), 53(5), 1489-1494

CODEN: JAFCAU; ISSN: 0021-8561

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Isohumulones, dihydroisohumulones, tetrahydroisohumulones, and humulinones, important hop-derived bittering compds. in beer, were shown to give rise to reactive triacylmethyl radicals on interaction with triplet-excited riboflavin after spin trapping by 5,5-dimethyl-1-pyrroline N-oxide or 2-methyl-2-nitrosopropane, followed by ESR spectroscopy combined with spectral simulation. Electron abstraction from the ionized β-tricarbonyl chromophore, which is common to all five-membered ring hop derivs., is the initial event on

photoinduced degradation Radicaloid decomposition of isohumulones leads to precursors for 3-methylbut-2-ene-1-thiol, the lightstruck constituent in beer. Interaction of reduced derivs. of isohumulones with triplet-excited riboflavin furnished radical precursors of volatile aldehydes, which may lead to the development of unpleasant stale or cardboard flavors.

REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 5 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN DUPLICATE 2

ACCESSION NUMBER: 2005:382489 CAPLUS

DOCUMENT NUMBER: 143:152217

TITLE: Fate of flavins in sensitized photodegradation of isohumulones and reduced derivatives: studies on formation of radicals via EPR combined with detailed product analyses

AUTHOR(S): Heyerick, Arne; Huvaere, Kevin; De Keukeleire, Denis; Forbes, Malcolm D. E.

CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, Laboratory of Pharmacognosy and Phytochemistry, Ghent University, Ghent, B-9000, Belg.

SOURCE: Photochemical & Photobiological Sciences (2005), 4(5), 412-419

CODEN: PPSHCB; ISSN: 1474-905X

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Photodegrdn. of isohumulones accounts for formation of the lightstruck flavor in beer. The reactions involved are mediated by riboflavin, a natural photosensitizer present in beer in ppb quantities. The results of an investigation of this sensitized degradation process are presented herein. Product analyses and ESR spectroscopy, in steady-state as well as in time-resolved mode, offer extensive insight into the photophys. and photochem. details of the degradation mechanism. In contrast to energy transfer and Norrish type I α -cleavage reactions that take place on direct irradiation of isohumulones, the sensitization pathway proceeds via one-electron redox chemical involving the excited triplet state of riboflavin and derivs. The flavin semiquinone radical thus formed could be readily detected, either by steady state or by time-resolved ESR spectroscopy. Superimposed signals in the spectra revealed the presence of radical fragments derived from isohumulones or tetrahydroisohumulones, which, on recombination with riboflavin semiquinone radicals, produced stable reaction products that were identified by HPLC-MS. However, no superimposed signals were observed on sensitized irradiation of dihydroisohumulones.

REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 6 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:1007048 CAPLUS

DOCUMENT NUMBER: 144:310653

TITLE: Shining light on the photodecomposition of beer

AUTHOR(S): Huvaere, Kevin; De Keukeleire, Denis

CORPORATE SOURCE: Ghent University, Chent, B-9000, Belg.

SOURCE: Spectrum (Bowling Green, OH, United States) (2005), 18(2), 18-24

CODEN: SBGOA7; ISSN: 1044-5536

PUBLISHER: Center for Photochemical Sciences

DOCUMENT TYPE: Journal; General Review

LANGUAGE: English

AB A review. Light exposure is harmful to the beer quality and protection is necessary against photodecompn. of hop-derived bitter compds. Prevention of the lightstruck flavor has mainly focused on phys. protection; for example, cans and dark-colored bottles should prevent light from

interacting with beer. Still, beer is prone to undergo photodecompn. during consumption from a glass and the final reaction product, 3-methylbut-2-ene-1-thiol, is the main cause of the so-called "skunky flavor". The studies have resulted in clear insights into the mechanisms that govern the photodecompn. of beer. Direct absorption of UV light by isohumulones and tetrahydroisohumulones leads to energy transfer from the excited triplet state of the enolized β -tricarboxyl chromophore to the α -hydroxyketo group, which subsequently undergoes α -cleavage to a 4-methylpent-3-enoyl radical and a 4-methylpentanoyl radical, resp. These radicals react further to unpleasant volatiles, such as 3-methylbut-2-ene-1-thiol, which arises by decarbonylation of the 4-methylpent-3-enoyl radical followed by trapping by a thiyl radical. Dihydroisohumulones lacking the α -hydroxyketo group can not undergo α -cleavage and the excitation energy is dissipated, hence, the compds. are lightstable, at least on direct irradiation

REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 7 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:936070 CAPLUS

DOCUMENT NUMBER: 141:400871

TITLE: Anti-inflammatory pharmaceutical compositions for reducing inflammation and the treatment or prevention of gastric toxicity

INVENTOR(S): Babish, John G.; Tripp, Matthew L.; Bland, Jeffrey S.; Howell, Terrence; Darland, Gary K.; Lerman, Robert H.; Lukaczer, Daniel O.

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 49 pp., Cont.-in-part of U.S. Ser. No. 689,856.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 7

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004219240	A1	20041104	US 2004-774048	20040205
US 2003008021	A1	20030109	US 2001-885721	20010620
US 2004086580	A1	20040506	US 2003-464410	20030618
US 2004115290	A1	20040617	US 2003-464834	20030618
US 2004151792	A1	20040805	US 2003-689856	20031020
AU 2004283065	A1	20050506	AU 2004-283065	20040521
CA 2526804	A1	20050506	CA 2004-2526804	20040521
WO 2005039483	A2	20050506	WO 2004-US16043	20040521
WO 2005039483	A3	20050929		
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EP 1626731	A2	20060222	EP 2004-809400	20040521
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR				
US 2006141081	A1	20060629	US 2006-355145	20060215
US 2006141082	A1	20060629	US 2006-355306	20060215
PRIORITY APPLN. INFO.:			US 2001-885721	A2 20010620

US 2002-420383P	P	20021021
US 2003-450237P	P	20030225
US 2003-400293	B2	20030326
US 2003-401283	B2	20030326
US 2003-472460P	P	20030522
US 2003-464410	A2	20030618
US 2003-464834	A2	20030618
US 2003-689856	A2	20031020
US 2004-774048	A	20040205
WO 2004-US16043	W	20040521

OTHER SOURCE(S): MARPAT 141:400871

AB The invention provides hops (*Humulus lupulus*) exts. or derivs. thereof, such as humulone, cohumulone, adhumulone, isohumulone, etc., for use in treating a patient prophylactically and/or therapeutically for ulcerogenic-type disorders of the stomach and/or intestines. The ulcerogenic disorders can be induced chemical, environmentally, by infection, and/or by stress. The invention also provides a pharmaceutical composition comprising an active amount of hops exts. or derivs. thereof, in combination with an analgesic compound and/or an anti-inflammatory compound. The invention further provides for use of hops exts. or derivs. thereof, significantly reducing and/or therapeutically treating ulcerogenic-type disorders of the stomach and/or intestines. For example, the hop preparation Redihop containing rho-iso- α -acids when combined with NSAIDs (ibuprofen and aspirin) not only attenuated the gastropathy of NSAIDs by decreasing an inhibition of PGE2 synthesis in AGS human gastric mucosal cells, but also increased therapeutic indexes of both ibuprofen and aspirin.

L13 ANSWER 8 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:633066 CAPLUS

DOCUMENT NUMBER: 141:179610

TITLE: pharmaceutical and nutraceutical compositions containing extracts from hop and rosemary for treatment and prevention of inflammatory-related disorders

INVENTOR(S): Tripp, Matthew L.; Babish, John G.; Bland, Jeffrey S.; Darland, Gary K.; Lerman, Robert; Lukaczer, Daniel O.; Liska, Deann J.; Howell, Terrence

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 66 pp., Cont.-in-part of U.S. Pat. Appl. 2004 86,580.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 7

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004151792	A1	20040805	US 2003-689856	20031020
US 2003008021	A1	20030109	US 2001-885721	20010620
US 2004086580	A1	20040506	US 2003-464410	20030618
US 2004115290	A1	20040617	US 2003-464834	20030618
US 2004219240	A1	20041104	US 2004-774048	20040205
AU 2004283065	A1	20050506	AU 2004-283065	20040521
CA 2526804	A1	20050506	CA 2004-2526804	20040521
WO 2005039483	A2	20050506	WO 2004-US16043	20040521
WO 2005039483	A3	20050929		

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RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,

AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

EP 1626731 A2 20060222 EP 2004-809400 20040521
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR
US 2007020352 A1 20070125 US 2006-326874 20060106
US 2006141081 A1 20060629 US 2006-355145 20060215
US 2006141082 A1 20060629 US 2006-355306 20060215
US 2006177531 A1 20060810 US 2006-403016 20060412

PRIORITY APPLN. INFO.:

US 2001-885721 A2 20010620
US 2002-420383P P 20021021
US 2003-450237P P 20030225
US 2003-400293 B2 20030326
US 2003-401283 B2 20030326
US 2003-464410 A2 20030618
US 2003-464834 A2 20030618
US 2003-472460P P 20030522
US 2003-689856 A2 20031020
US 2004-774048 A 20040205
WO 2004-US16043 W 20040521
US 2004-866315 B2 20040610

OTHER SOURCE(S): MARPAT 141:179610

AB A natural formulation of compds. that would to modulate inflammation is disclosed. The formulation would also inhibit expression of COX-2, inhibit synthesis of prostaglandins selectively in target cells, and inhibit inflammatory response selectively in target cells. The compns. containing at least one fraction isolated or derived from hops. Other embodiments relate to combinations of components, including at least one fraction isolated or derived from hops, tryptanthrin and conjugates thereof, rosemary, an extract or compound derived from rosemary, a triterpene species, or a diterpene lactone or derivs. or conjugates thereof. For example, an oral dietary supplement containing isocohumulone, dihydroadhumulone, tetrahydroisocohumulone, hexahydroisohumulone from rosemary was found to be able to normalization the joint function after two to ten doses.

L13 ANSWER 9 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:493479 CAPLUS

DOCUMENT NUMBER: 141:33790

TITLE: Modulation of inflammation by hops fractions and derivatives

INVENTOR(S): Tripp, Matthew L.; Babish, John G.; Bland, Jeffrey S.; Darland, Gary K.; Lerman, Robert; Lukaczer, Daniel O.; Liska, DeAnn J.; Howell, Terrence

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 26 pp., Cont.-in-part of US Ser. No. 400,293, abandoned.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 7

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004115290	A1	20040617	US 2003-464834	20030618
US 2003008021	A1	20030109	US 2001-885721	20010620
CA 2503196	A1	20040506	CA 2003-2503196	20031020
WO 2004037180	A2	20040506	WO 2003-US33362	20031020
WO 2004037180	A3	20040930		

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RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

AU 2003286549 A1 20040513 AU 2003-286549 20031020
US 2004151792 A1 20040805 US 2003-689856 20031020
EP 1558271 A2 20050803 EP 2003-777751 20031020

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JP 2006508182 T 20060309 JP 2005-501640 20031020
NZ 539642 A 20070126 NZ 2003-539642 20031020
US 2004219240 A1 20041104 US 2004-774048 20040205
AU 2004283065 A1 20050506 AU 2004-283065 20040521
CA 2526804 A1 20050506 CA 2004-2526804 20040521
WO 2005039483 A2 20050506 WO 2004-US16043 20040521
WO 2005039483 A3 20050929

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EP 1626731 A2 20060222 EP 2004-809400 20040521

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR

US 2007020352 A1 20070125 US 2006-326874 20060106
US 2006127511 A1 20060615 US 2006-344552 20060130
US 2006127512 A1 20060615 US 2006-344554 20060130
US 2006127516 A1 20060615 US 2006-344559 20060130
US 2006141081 A1 20060629 US 2006-355145 20060215
US 2006141082 A1 20060629 US 2006-355306 20060215
US 2006177531 A1 20060810 US 2006-403016 20060412
US 2006193933 A1 20060831 US 2006-403034 20060412

PRIORITY APPLN. INFO.: US 2001-885721 A2 20010620
US 2002-420383P P 20021021
US 2003-450237P P 20030225
US 2003-400293 B2 20030326
US 2003-401283 B2 20030326
US 2003-472460P P 20030522
US 2003-464410 A 20030618
US 2003-464834 A 20030618
US 2003-689856 A2 20031020
WO 2003-US33362 W 20031020
US 2004-774048 A 20040205
WO 2004-US16043 W 20040521
US 2004-866315 B2 20040610

OTHER SOURCE(S): MARPAT 141:33790

AB A natural formulation of compds. for the modulation of inflammation is disclosed. The formulation would also inhibit expression of COX-2, inhibit synthesis of prostaglandins selectively in target cells, and inhibit inflammatory response selectively in target cells. The compns. contain at least one fraction isolated or derived from hops.

DOCUMENT NUMBER: 140:368679
 TITLE: Synergistic compositions that treat or inhibit pathological conditions associated with inflammatory response
 INVENTOR(S): Tripp, Matthew L.; Babish, John G.; Bland, Jeffrey S.; Darland, Gary K.; Lerman, Robert; Lukaczer, Daniel O.; Liska, Deann J.; Howell, Terrence
 PATENT ASSIGNEE(S): USA
 SOURCE: U.S. Pat. Appl. Publ., 41 pp., Cont.-in-part of U.S. Ser. No. 400,293, abandoned.
 CODEN: USXXCO
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 7
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004086580	A1	20040506	US 2003-464410	20030618
CA 2503196	A1	20040506	CA 2003-2503196	20031020
WO 2004037180	A2	20040506	WO 2003-US33362	20031020
WO 2004037180	A3	20040930		
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RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2003286549	A1	20040513	AU 2003-286549	20031020
US 2004151792	A1	20040805	US 2003-689856	20031020
EP 1558271	A2	20050803	EP 2003-777751	20031020
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JP 2006508182	T	20060309	JP 2005-501640	20031020
NZ 539642	A	20070126	NZ 2003-539642	20031020
US 2004219240	A1	20041104	US 2004-774048	20040205
AU 2004283065	A1	20050506	AU 2004-283065	20040521
CA 2526804	A1	20050506	CA 2004-2526804	20040521
WO 2005039483	A2	20050506	WO 2004-US16043	20040521
WO 2005039483	A3	20050929		
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EP 1626731	A2	20060222	EP 2004-809400	20040521
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR				
US 2007020352	A1	20070125	US 2006-326874	20060106
US 2006127513	A1	20060615	US 2006-344555	20060130
US 2006127514	A1	20060615	US 2006-344556	20060130
US 2006127515	A1	20060615	US 2006-344557	20060130
US 2006127517	A1	20060615	US 2006-344561	20060130
US 2006141081	A1	20060629	US 2006-355145	20060215

US 2006141082	A1	20060629	US 2006-355306	20060215
US 2006177531	A1	20060810	US 2006-403016	20060412
PRIORITY APPLN. INFO.:			US 2002-420383P	P 20021021
			US 2003-450237P	P 20030225
			US 2003-400293	B2 20030326
			US 2003-401283	B2 20030326
			US 2001-885721	A2 20010620
			US 2003-472460P	P 20030522
			US 2003-464410	A 20030618
			US 2003-464834	A 20030618
			US 2003-689856	A2 20031020
			WO 2003-US33362	W 20031020
			US 2004-774048	A 20040205
			WO 2004-US16043	W 20040521
			US 2004-866315	B2 20040610

OTHER SOURCE(S): MARPAT 140:368679

AB A natural formulation of compds. that would modulate inflammation is disclosed. The formulation would also inhibit expression of COX-2, inhibit synthesis of prostaglandins selectively in target cells, and inhibit inflammatory response selectively in target cells. The compns. contains at least one fraction isolated or derived from hops. Other embodiments relate to combinations of components, including at least one fraction isolated or derived from hops, tryptanthrin and conjugates thereof, rosemary, an extract or compound derived from rosemary, a triterpene species, or a diterpene lactone or derivs. or conjugates thereof. For example, a synergistic inhibition of PGE2 synthesis in target cells by hop CO2 extract containing 30 to 60% alpha-acids and 15 to 45% beta-acids in combination with triterpenoids oleanolic acid and ursolic acid was exhibited.

L13 ANSWER 11 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:722575 CAPLUS

DOCUMENT NUMBER: 142:5781

TITLE: Photooxidative degradation of beer bittering principles: product analysis with respect to lightstruck flavor formation

AUTHOR(S): Huvaere, Kevin; Sinnaeve, Bart; Van Bocxlaer, Jan; De Keukeleire, Denis

CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, Laboratory of Pharmacognosy and Phytochemistry, Ghent University, Ghent, B-9000, Belg.

SOURCE: Photochemical & Photobiological Sciences (2004), 3(9), 854-858

CODEN: PPSHCB; ISSN: 1474-905X

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Isohumulones, the main bittering agents in beer, are decomposed by light-induced reactions, thereby leading to radical precursors on the pathway to lightstruck flavor formation. Excited flavins, formed on visible-light irradiation, readily interact with isohumulones, as well as with reduced and oxidized derivs. thereof. From identification of both volatile and non-volatile reaction products thus formed, feasible degradation mechanisms are proposed.

REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 12 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:257951 CAPLUS

DOCUMENT NUMBER: 141:53110

TITLE: Riboflavin-sensitized photooxidation of isohumulones and derivatives

AUTHOR(S): Huvaere, Kevin; Olsen, Karsten; Andersen, Mogens L.; Skibsted, Leif H.; Heyerick, Arne; De Keukeleire,

CORPORATE SOURCE: Denis
Faculty of Pharmaceutical Sciences, Laboratory of
Pharmacognosy and Phytochemistry, Ghent University,
Ghent, B-9000, Belg.
SOURCE: Photochemical & Photobiological Sciences (2004), 3(4),
337-340
CODEN: PPSHCB; ISSN: 1474-905X
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Isohumulones, the bitter principles in beer, are decomposed by
light-induced reactions, thereby adversely affecting beer quality. Upon
absorption of visible light, riboflavin is excited and interacts with
isohumulones, as well as with oxidized and reduced derivs.
thereof. Reaction kinetics were investigated by laser flash photolysis at
355 nm and at 440 nm, and anal. of kinetic data afforded detailed insights
into the reaction mechanism.
REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 13 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN DUPLICATE 3
ACCESSION NUMBER: 2004:285087 CAPLUS
DOCUMENT NUMBER: 141:37710
TITLE: Analysis of iso- α -acids and reduced
iso- α -acids in beer by direct injection and
liquid chromatography with ultraviolet absorbance
detection or with mass spectrometry
AUTHOR(S): Vanhoenacker, G.; De Keukeleire, D.; Sandra, P.
CORPORATE SOURCE: Research Institute for Chromatography, Kortrijk,
B-8500, Belg.
SOURCE: Journal of Chromatography, A (2004), 1035(1), 53-61
CODEN: JCRAEY; ISSN: 0021-9673
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A liquid chromatog. (LC) method is described for the simultaneous anal. of
iso- α -acids and reduced iso- α -acids in beer. Volatile mobile
phase additives were selected to enable hyphenation to mass spectrometric
(MS) operated in the atmospheric pressure chemical ionization (APCI) mode.
Contrary
to other recent LC optimization procedures for the same compds., an alkaline
pH was selected, thereby improving peak shape and selectivity. Both UV
and MS detection are sensitive enough to analyze beers without sample
pre-concentration All major bitter acids are separated within 65 min with
exception
of cis-dihydroisoadhumulone, which co-elutes with trans-
isocohumulone. Due to the selectivity of the MS, these compds.
could be differentiated according to their m/z value. The performance in
terms of quantification of bitter acids by LC-UV and LC-MS are compared
for standard solns. and a selection of 14 beers.
REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 14 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN DUPLICATE 4
ACCESSION NUMBER: 2003:828724 CAPLUS
DOCUMENT NUMBER: 140:27056
TITLE: Radicaloid-type oxidative decomposition of beer
bittering agents revealed
AUTHOR(S): Huvaere, Kevin; Andersen, Mogens L.; Olsen, Karsten;
Skibsted, Leif H.; Heyerick, Arne; De Keukeleire,
Denis
CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, Laboratory of
Pharmacognosy and Phytochemistry, Ghent University,
Ghent, 9000, Belg.

SOURCE: Chemistry--A European Journal (2003), 9(19), 4693-4699
CODEN: CEUJED; ISSN: 0947-6539
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Trans-Isohumulones, dihydroisohumulones, tetrahydroisohumulones, and humulinones, which are important hop-derived flavor components of beer, were found, by using electrolysis of organic solns., to be stable against oxidation, like weak acids; however, they are readily oxidized in their anionic forms as present in beer. Oxygen- and carbon-centered radicals were formed by oxidation and identified by using spin trapping under aerobic and anaerobic conditions, followed by EPR (ESR) spectroscopy. Generated radicals were reactive, most likely degrading into products lacking the tricarbonyl chromophore; this is typical of five-membered-ring hop derivs. Thus, flavor-active beer constituents may degrade oxidatively in the absence of oxygen, thereby leading to reaction products that escape UV detection.
REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 15 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN DUPLICATE 5
ACCESSION NUMBER: 1999:194941 CAPLUS
DOCUMENT NUMBER: 131:43732
TITLE: Investigation of hop and beer bitter acids by coupling of high-performance liquid chromatography to nuclear magnetic resonance spectroscopy
AUTHOR(S): Pusecker, K.; Albert, K.; Bayer, E.
CORPORATE SOURCE: Institute of Organic Chemistry, Research Center for Nucleic Acid and Peptide Chemistry, University of Tübingen, Tübingen, D-72076, Germany
SOURCE: Journal of Chromatography, A (1999), 836(2), 245-252
CODEN: JCRAEY; ISSN: 0021-9673
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
AB HPLC-NMR coupling is becoming used for various applications, including the anal. of natural products. Its great potential is demonstrated by the anal. of hop bitter acids, such as humulones, isohumulones, dihydroisohumulones and tetrahydroisohumulones, using online and stopped-flow techniques. 1H-NMR and 2-dimensional NMR spectra recorded for all hop bitter acids allowed unambiguous identification. It is shown, that hyphenation of HPLC and NMR spectroscopy offers unique opportunities for anal. and quality control of hops and beer.
REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 16 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 1998:497010 CAPLUS
DOCUMENT NUMBER: 129:215965
TITLE: Natural foam stabilizing and bittering compounds derived from hops
AUTHOR(S): Smith, Robert J.; Davidson, Darwin; Wilson, Richard J. J.
CORPORATE SOURCE: S. S. Steiner, Inc., Yakima, WA, 98909, USA
SOURCE: Journal of the American Society of Brewing Chemists (1998), 56(2), 52-57
CODEN: JSBCD3; ISSN: 0361-0470
PUBLISHER: American Society of Brewing Chemists, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Various naturally occurring hop resin acids were isolated by preparative HPLC from hops and hop products. Two resin acids were tentatively identified by NMR techniques to be the minor constituent α -acids, adprehumulone and prehumulone. The isomerized derivative of the former

considerably improved the foam stability and lacing of a com. brand of beer. Dihydro- α -acids have previously been shown to occur and form in hops and hop products. Two of the dihydroiso- α -acids were isolated by preparative HPLC, and dihydroisohumulone was shown to substantially improve foam stability and lacing of various brands of com. beer as compared with the iso- α -acids.

REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 17 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1959:67838 CAPLUS

DOCUMENT NUMBER: 53:67838

ORIGINAL REFERENCE NO.: 53:12331i,12332a-i,12333a-b

TITLE: Chemistry of hop constituents. XIII. Hydrogenation of isohumulone

AUTHOR(S): Brown, P. Margaret; Howard, G. A.; Tatchell, A. R.

CORPORATE SOURCE: Brewing Ind. Research Foundation, Nutfield, UK

SOURCE: Journal of the Chemical Society (1959) 545-51

CODEN: JCSOA9; ISSN: 0368-1769

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. C.A. 52, 12818e. Isohumulone A (I) (853 mg.) was hydrogenated in 70 ml. MeOH over 100 mg. PtO₂; extraction with Et₂O followed by aqueous Na₂CO₃ and distillation at 130° and 2 + 10⁻³ mm. gave neohydroisohumulone (II), λ 253 m μ (ϵ 11,800) and 274 m μ (ϵ 17,200) in acidic and alkaline EtOH, resp. II tasted bitter, gave no copper complex soluble in CHCl₃, failed to reduce Fehling solution, gave a pos. CHI₃ reaction, and was unaffected by boiling N alc. alkali or by KHSO₄ in boiling PhMe. A trace of Me₂CO was produced with O₃. Similar hydrogenation of 2 crystalline isohumulones [m. 123-4°, [α]D -15.7° (MeOH); m. 129-30°, [α]D -40.6° (MeOH)] gave products which failed to distil at 135° and 10⁻⁴ mm., but which had ultraviolet light absorption identical to that of II. Hydrogenation of 617 mg. I in 20 ml. HOAc over PtO₂ gave 70% tetrahydroisohumulone (III), m. 31-3°. Hydrogenation of 616 mg. I in 5 ml. MeOH and 50 ml. aqueous 2N Na₂CO₃ over PtO₂ gave dihydroisohumulone A (IV), λ 225 and 274 m μ (E₁%1cm. 310 and 256) in acidic EtOH and 252 and 272 (inflection) m μ (E₁%1cm. 405 and 350) in alkaline EtOH, iodine value 72, after chromatographic separation

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silica gel using C₆H₆. IV gave a brown color with FeCl₃ in MeOH. Hydrolysis with aqueous alkali in EtOH gave γ -methylvaleric acid (V) in low yield. Hydrogenation over Pd-C gave a product with ultraviolet light absorption like that of I, III, and IV. Trituration with C₆H₆ gave a soluble and an insol. fraction in the proportion 2:1, both of which gave dihydrohumulinic acid (VI) and V after alkaline hydrolysis. Countercurrent distribution of the soluble and insol. fractions gave 71 and 50% III, resp., m. 32-4°, [α]D 24.5° and 98° in neutral and alkaline MeOH, resp., λ 230 and 275 m μ (E₁%1cm. 250 and 250) and 253 m μ (E₁%1cm. 456) in acidic and alkaline EtOH, resp. Hydrogenation of III in MeOH over PtO₂ gave II. III had a partition coefficient 1.04 in Me₂CHAm-phosphate buffer (0.5M; pH 6.5). Hydrogenation of IV in MeOH over PtO₂ gave II. Reduction over PtO₂ in HOAc gave an oil which distilled at 120° and 5 + 10⁻⁴ mm. to give a product with ultraviolet spectrum like that of II but with different infrared absorption and countercurrent distribution. Hydrogenation of 892 mg. isocohumulone A (VII) in 30 ml. HOAc over PtO₂ gave 85% tetrahydroisocohumulone (VIII), m. 40-6°, after chromatography over silica gel with C₆H₆ and distillation at 145° and 10⁻³ mm., [α]D 28° and 89° in MeOH and alkaline MeOH, resp., partition coefficient 0.47 in the system listed above, λ 230 and 273 m μ (E₁%1cm. 294 and 274) and 253 m μ (E₁%1cm. 515) in acidic and alkaline EtOH. Tetrahydrocophumulone (IX) (480 mg.) in 74 ml. aqueous 1/15N NaOH was refluxed 9 min. under N. Purification by chromatography gave VIII, m. 55-8°

(distilled at 105° and 10-5 mm.), partition coefficient 0.46, $[\alpha]_D^{20}$ 6° in neutral and alkaline EtOH, λ 230 and 273 m μ (E1%1cm. 264 and 284) and 253 m μ (E1%1cm. 494) in acidic and alkaline EtOH. \pm -IX (2.86 g.) was isomerized in alkaline EtOH to give 1.47 g. VIII, m. 50-2°, λ 230 and 273 m μ (E1%1cm. 260 and 250) and 253 m μ (E1%1cm. 574) in acidic and alkaline EtOH. Refluxing 668 mg. IX with 100 ml. 1/15N NaOH under N 9 min. gave III, m. 49-53°, $[\alpha]_D^{20}$ -2 and +13° in neutral and alkaline MeOH, partition coefficient 1.04, λ 230 and 275 m μ (E1%1cm. 250 and 265) and 253 m μ (E1%1cm. 480) in acidic and alkaline EtOH. Hydrolysis of 478 mg. III with 7 ml. N NaOH and 3 ml. EtOH at reflux under N 3 hrs. and purification gave V and VI. Oxidation of 200 mg. III in 10 ml. refluxing HOAc with 450 mg. Bi2O3 5 hrs. gave isohumulonic acid, m. 142-3°. III was unchanged by boiling HOAc alone. Similar oxidation of VIII gave isocohumulonic acid, m. 121-3°. Similar oxidation of I gave 5-(3-methylbut-2-enyl)-3-isovaleryl cyclopentane-1,2,4-trione. Oxidation of 500 mg. II under reflux in 14 ml. 2N NaOH and 5 ml. 30% H2O2 gave 370 mg. of an oil on purification which in turn gave the p-bromophenacyl derivative of V, m. 75-7°. In a similar experiment, 98% the oxidation product was soluble in aqueous NaHCO3; gas chromatography gave 6:78:17 V, isovaleric acid, and a C7 acid. After steam distillation of the mixture, the nonvolatile acids were again oxidized with H2O2 in boiling alkali; 22% V resulted. Thus, oxidation of II finally gave about 1.9 moles V. Humulone (6.37 g.) was isomerized by Carson's method (C.A. 47, 9926b) to give 729 mg. "isohumulone," m. 124-5°, $[\alpha]_D^{20}$ 10.4 and 56° in neutral and alkaline MeOH, resp. Countercurrent distribution with Me2CHAm and phosphate-citrate buffer (pH 5.0) indicated one major component with partition coefficient 1.42, together with minor components. The residue remaining after removal of the "isohumulone" was dissolved in Et2O and shaken with 2N NaOH, and the insol. Na salt was removed. The oily isohumulone (3.8 g.) obtained contained 75% I. Hydrogenation of a portion of I over PtO2 in HOAc gave 60% III. Neohydroisocohumulone (867 mg.) in 20 ml. EtOH was treated overnight with 1.2 g. NaIO4 in 20 ml. H2O. After purification the residue was treated with alc. 2,4-dinitrophenylhydrazine-HCl and the hydrazones examined chromatographically on Al2O3. No low mol. weight ketones were observed. The alkaline solution after hydrolysis gave V. Infrared spectral data is given for a number of the compds. and structures are postulated for II, III, and IV, and the isohumulonic acids.

L13 ANSWER 18 OF 18 CAPLUS COPYRIGHT 2007 ACS on STN

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DOCUMENT NUMBER: 47:58556
ORIGINAL REFERENCE NO.: 47:9926b-i,9927a-c
TITLE: The alkaline isomerization of humulone
AUTHOR(S): Carson, J. F.
CORPORATE SOURCE: Western Regional Research Labs., Albany, CA
SOURCE: Journal of the American Chemical Society (1952), 74, 4615-20
CODEN: JACSAT; ISSN: 0002-7863

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GI For diagram(s), see printed CA Issue.

AB The isomerization of humulone (an antibiotic and flavoring component of hops) in alkaline MeOH yields complex mixts. from which 3 crystalline isomers of

humulone have been isolated in small yield. On the basis of their chemical reactions and absorption spectra, 2 of the compds. are best represented by structure I and the 3rd by structure II. The compds. are designated (+)-isohumulone (III), (-)-isohumulone (IV), and inactive isohumulone (II). II, III, and IV differ from humulone (V) in that they do not form insol. Pb salts or crystalline o-C6H4-(NH2)2 complexes. As with V, no crystalline derivs. could be prepared utilizing the CO, enolic,

or

tertiary OH functions. Attempts to prepare Me esters with CH_2N_2 , acetates, phenylurethans, oximes, semicarbazones, or 2,4-dinitrophenylhydrazones yielded only oils or resins. The crystalline isomers give a red color with FeCl_3 ; they are not bitter in alc. or aqueous solution. In addition to the crystalline

isomers, an oil was isolated from the mother liquor and separated into 2 noncrystg. components and a crystalline fraction. The 2 oily fractions are intensely bitter; their structures have not been elucidated, but on the basis of the chemical reactions and absorption spectra they appear to be closely related to the crystalline isomers. Alkaline degradation of the 3 crystalline

isomers in each case yields optically inactive humulinic acid (VI) and a mixture of Me_2CO and Me_2CHCHO . V (162 g.) in 1 l. MeOH was neutralized to phenolphthalein with KOH, 9.2 g. KOH added, the solution made up with MeOH to 2 l., refluxed 3 hrs., cooled to 20° , acidified with 20 cc. 5N HCl, concentrated in vacuo below 25° to 400 cc., treated with 600 cc. N HCl, the resulting resinous suspension extracted 3 times with 400-cc. portions Et₂O, the combined Et₂O exts. were washed with H₂O, dried, and concentrated in vacuo, and the residue diluted with 400 cc. petr. ether to give 24.8 g. (15%) yellow crystalline product, separated by fractional crystallization into

2.2 g. III,

m. $133-4^\circ$, $[\alpha]_{\text{D}26} 112^\circ$ (MeOH), and 1.14 g. IV, m. $134-5^\circ$, $[\alpha]_{\text{D}25} -60.6$ (EtOAc). From the mother liquor from III and IV was isolated by chromatography on silicic acid and fluorescent ZnS 580 mg. II, m. $145-6^\circ$, $[\alpha]_{\text{D}26} 0^\circ$ (EtOAc or CHCl_3).

From the oily mother liquor from II was isolated by chromatography a yellow oil, $[\alpha]_{\text{D}} 17.7^\circ$ (EtOAc), $\lambda_{\text{maximum}} 253 \text{ m}\mu$ E1% 435; a yellow oil, $[\alpha]_{\text{D}} 50.3^\circ$ (EtOAc), $\lambda_{\text{maximum}} 253 \text{ m}\mu$, E1% 461; and a yellow crystalline solid, m. $133-3.6^\circ$ (from petr. ether), optically inactive. III (0.5 g.) in 45 cc. MeOH shaken 10 min. with H at 26° and 760 mm. in the presence of 185 mg. Pd-C yielded 360 mg. (72%) (+)-dihydroisohumulone (VII), m. $147-8^\circ$

(from aqueous MeOH), $[\alpha]_{\text{D}26} 72.4 \pm 1.0^\circ$ (EtOAc). Similarly was prepared the (-)-dihydroisohumulone (VIII), m. $162-5^\circ$ (from aqueous MeOH), $[\alpha]_{\text{D}26} -76.9 \pm 1.0^\circ$ (EtOAc). IV (540

mg.) in 15 cc. absolute EtOH and 50 cc. N KOH was refluxed 2 hrs. and the solution concentrated by distillation to 1/3 its original volume and acidified to yield 183

mg. (46%) VI, m. $93-4^\circ$; the distillate from the reaction mixture was collected in 50 cc. 3N HCl containing 1 g. 2,4-(O₂N)₂C₆H₄NHNH₂, the mixture extracted 4 times with 50-cc. portions of C₆H₆, and the extract dried,

concentrated to

50 cc., diluted with 100 cc. petr. ether, and chromatographed on silicic acid to give 180 mg. (48 mole-%) isobutyraldehyde 2,4-dinitrophenylhydrazone, m. $184-6^\circ$, and 40 mg. (11 mole-%) Me_2CO 2,4-dinitrophenylhydrazone (IX), m. $125-6^\circ$. Similar alkaline degradation of VIII gave dihydrohumulinic acid, m. $124.8-5.4^\circ$ (from C₆H₁₄). Ozonized O was passed 0.5 hr. at $19-20^\circ$ through 337 mg. IV in 30 cc. AcOH, the mixture decomposed with 100 cc. H₂O and 5 g. Zn dust, filtered, and the filtrate distilled; from the distillate was isolated 210 mg. (93 mole-%) IX, and 12 mg. 2,4-dinitrophenylhydrazone of EtAc. Similar degradation of the oily fractions gave in addition to IX small yields (9-14%) of 2,4-dinitrophenylhydrazone of Me_2CHCHO . II, III, IV, VI, and etude isohumulone oil were all bacteriostatically inactive at 0.1% for *Escherichia coli*, *Micrococcus conglomeratus*, *M. pyogenes* var. *aureus*, *Sarcina lutea*, and *Mycobacterium tuberculosis* var. *hominis* by an agar streak method.